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THE BRITTLE FRACTURE OF AMORPHOUS THERMOPLASTIC POLYMERS

BY

A. T. DiBENEDETTO  
K. L. TRACHTE

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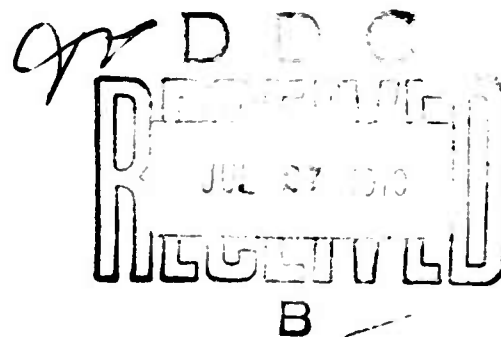
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ROLF BUCHDAHL

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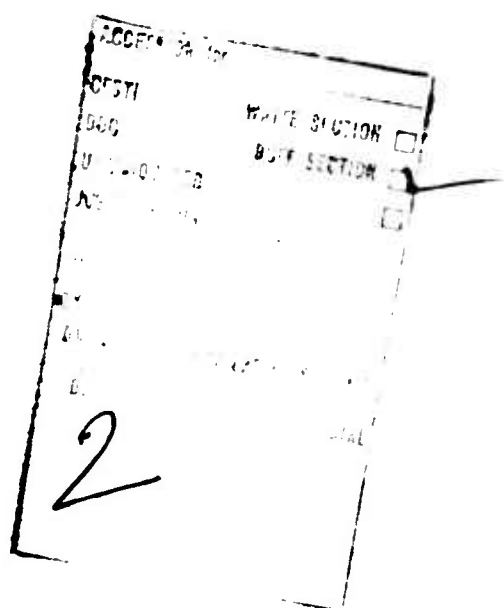
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A. T. DiBENEDETTO  
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FEBRUARY 1970

MONSANTO/WASHINGTON UNIVERSITY ASSOCIATION  
HIGH PERFORMANCE COMPOSITES PROGRAM  
SPONSORED BY ONR AND ARPA  
CONTRACT NO. N00014-67-C-0218, ARPA ORDER 876  
ROLF BUCHDAHL, PROGRAM MANAGER

MONSANTO RESEARCH CORPORATION  
800 NORTH LINDBERGH BOULEVARD  
ST. LOUIS, MISSOURI 63166

## FOREWORD

The research reported herein was conducted by the staff of the Monsanto/Washington University Association under the sponsorship of the Advanced Research Projects Agency, Department of Defense, through a contract with the Office of Naval Research, N00014-67-C-0218 (formerly N00014-66-C-0045), ARPA Order No. 876, ONR contract authority NR 356-484/4-13-66, entitled "Development of High Performance Composites."

The prime contractor is Monsanto Research Corporation. The Program Manager is Dr. Rolf Buchdahl (Phone—314-694-4721).

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# THE BRITTLE FRACTURE OF AMORPHOUS THERMOPLASTIC POLYMERS

A. T. DiBenedetto  
K. L. Trachte\*  
Materials Research Laboratory  
Washington University  
St. Louis, Missouri

## Abstract

The brittle fracture properties of polyphenylene oxide, polysulfone, polycarbonate, and polymethylmethacrylate thermoplastic polymers were investigated over a wide range of temperatures. Fracture energy measurements were made using double edge-notched tensile samples. Tensile strength, tensile strain, and initial elastic modulus were measured for calculation of the fracture energy and further analysis of the polymer behavior.

It was found that mechanical transitions in the tensile properties corresponded reasonably well with transitions in the fracture energy in the temperature range investigated. Fracture surface photographs permitted visual analysis of the fracture process. It was found that the roughest fracture surface corresponded to the maximum in the fracture energy for a given polymer.

A theory for prediction of polymer tensile yield strain is presented, based on the volume dilation concept. The implications of this theory are discussed in terms of the crack tip flow process leading to brittle fracture.

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\*Presently at the Esso Research Corporation, Baytown, Texas.

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## Introduction

There is substantial evidence in the literature that viscous flow or plastic deformation occurs near the tip of an advancing crack in glassy amorphous polymers. The measured fracture energy for glassy polymers is of the order of 1,000 times greater than the theoretical surface energy of the solid. This increase has been attributed to the energy requirements for viscous flow and polymer crazing at the crack tip.

It is reasonable to expect that, as plastic deformation becomes more inhibited by successive hindrance of the side group and main chain motions of the polymer molecule, the fracture energy should decrease toward the theoretical surface energy, which involves only breaking of atomic bonds.

Unexpectedly, it has been found that for polymethylmethacrylate (Plexiglas II UVA), the fracture energy continues to increase with decreasing temperature, at least to  $-60^{\circ}\text{F}$ . A three-fold increase in fracture energy was found as the test temperature was reduced from the main glass temperature,  $T_g$ , at  $220^{\circ}\text{F}$  to  $-60^{\circ}\text{F}$ . This data is in agreement with previous work [1, 2] on the same grade polymer. This is surprising in that one would expect plastic deformation to be already considerably reduced at this lower temperature, which is about  $260^{\circ}\text{F}$  below the main glass temperature.

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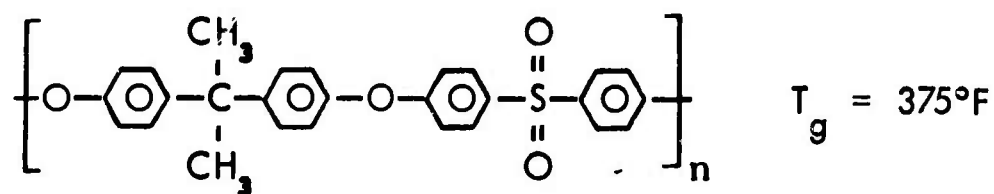
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Since it was presumed that a maximum in the fracture energy must occur somewhere between the main glass temperature and absolute zero of temperature, three amorphous polymers with high main glass temperatures were investigated, allowing a wide temperature range for testing. The four polymers investigated were:

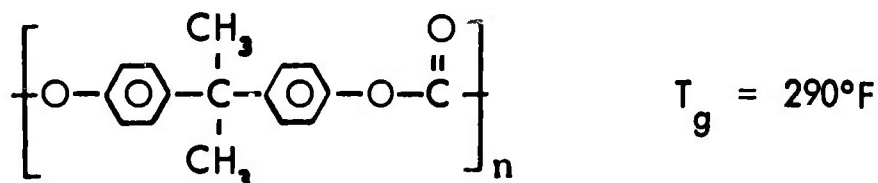
1. Polyphenylene oxide (General Electric Grade 631-111)



2. Polysulfone (Union Carbide Grade 1700)



3. Polycarbonate (General Electric "Lefan")



4. Polymethylmethacrylate (Rohm and Haas Plexiglas II UVA)



### Theory

The fracture mechanics of Irwin [3] was used to calculate the plane strain fracture energy,  $\gamma$ , required for catastrophic failure of the polymers. The sample geometry



used is shown in Figure 1. An iterative technique utilizing a computer was used to solve Irwin's approximation for the chosen geometry:

$$K_{I_c} \approx \sigma_n \left\{ W \left[ \tan \frac{\pi (a + K_{I_c}^2 / 2\pi \sigma_y^2)}{W} + .1 \sin \frac{2\pi (a + K_{I_c}^2 / 2\pi \sigma_y^2)}{W} \right] \right\}^{1/2} \quad (1)$$

where  $K_{I_c}$  = critical stress intensity parameter at the crack tip;  $\sigma_n$  = brittle tensile strength of a notched sample, based on the gross cross-sectional area;  $W$  = sample width;  $a$  = half-crack length at fracture;  $\sigma_y$  = tensile yield strength at the notch tip. The plane strain fracture energy  $\gamma$  was calculated from:

$$\gamma = K_{I_c}^2 (1 - \nu^2) / 2E \quad (2)$$

where  $\nu$  = Poisson's ratio of the polymer (taken as .35 for the four polymers); and  $E$  = initial elastic modulus of the polymer.

From the notched sample, the experimental values of  $W$ ,  $\sigma_n$ , and  $a$  were obtained. The brittle tensile strength,  $\sigma_n$ , was calculated directly from the load-deformation curve for the sample. The value of  $a$ , which includes the initial machined notch length and the slow crack growth prior to failure, was measured from the fractured sample surface to the nearest  $\pm .003$  cm under a microscope.

The values of  $\sigma_y$  used in the calculation of  $\gamma$  were obtained from a standard ASTM Tensile test D1708 [4]. A three-inch gauge length was used with all other dimensions as specified by ASTM. The strain rates used in our testing program were .013/min for the tensile tests and .25/min for the notched sample tests. The strain rate at the

crack tip, however, is likely to be much higher than that of the applied strain rate [5]. Since polymer properties are strain rate sensitive, the value of  $\sigma_y$  obtained from the tensile test is expected to be lower than the actual yield strength at the crack tip. However, an error analysis based on Equation (1) indicates that this uncertainty in  $\sigma_y$  does not significantly affect the calculated fracture energy within a three-decade range of strain rates [6].

The values of initial elastic modulus,  $E$ , used in the calculations were those obtained from the unnotched tensile tests.

The test temperature was varied from  $-60^\circ\text{F}$  to the main glass transition temperature of the polymer. The sample preparation and testing procedures have been discussed in more detail elsewhere [6, 7].

### Experimental Results

The effect of temperature on the energy required for polymer brittle fracture is shown in Figure 2. A maximum in  $\gamma$  was observed for polyphenylene oxide and polysulfone about  $150^\circ\text{F}$  below the main glass transition temperature. Our testing procedure did not allow for thick enough samples of polycarbonate to be tested to keep the fracture in a plane strain mode; consequently, some macroscopic yielding was observed around the fracture plane above  $80^\circ\text{F}$ . This is shown in Figure 3. Gross yielding at the notched section resulted in high values of  $\gamma$ , which are not reported with the plane strain data. The plane strain fracture data obtained for polycarbonate are similar to that of polyphenylene oxide and polysulfone.

The degree of roughness of the fracture surface is a qualitative measure of the fracture energy. For instance, the maxima in fracture energy for polyphenylene oxide and polysulfone occur at 250°F and 220°F, respectively. This is also the temperature region of maximum roughness, as seen from Figures 4 and 5. Polymethylmethacrylate exhibits a continuing increase in  $\gamma$  with decreasing temperature, with a corresponding increase in surface roughness as can be seen in Figure 6. As the test temperature approached the main glass temperature (220°F) for this polymer, a near perfect cleavage surface was formed (219°F). A temperature increase of only a few degrees completely changed the fracture mode. At 221°F, gross yielding occurred, resulting in shear failure at the notched section. This same phenomenon was observed with polysulfone and polyphenylene oxide just above  $T_g$ .

Over the past few years there have been numerous attempts to relate impact strength to secondary glass transitions. Perhaps the best survey to date is that of Boyer [8]. A comparison of the observed temperature response of  $\gamma$  with known secondary transitions for the four polymers [9-13], showed no apparent correlation between fracture energy and secondary transitions. In fact, the data did not reflect any change in  $\gamma$  at any of the secondary glass transitions.

For this reason, we concluded that the maximum in the plane strain fracture energy for polysulfone, polyphenylene oxide, and presumably polycarbonate is a manifestation of the approaching main glass temperature. This is supported by the tensile yield strength, yield strain, and initial elastic modulus data for the four polymers, as shown in Figures 7, 8 and 9. As the test temperature is lowered from the main glass

transition, the fracture energy increases for all four polymers. The tensile yield strength, yield strain, and initial elastic modulus also increase, at the same rate for all four polymers. Between  $T_g - 50^\circ\text{F}$  and  $T_g - 100^\circ\text{F}$ , the rate of increase in the tensile properties decreases abruptly for polyphenylene oxide, polysulfone, and polycarbonate. Considering the difference in strain rates between the tensile and notched tests, this mechanical transition compares reasonably well with the temperature,  $T_g - 150^\circ\text{F}$ , at which the maximum in  $\gamma$  is observed for polyphenylene oxide and polysulfone (and presumably polycarbonate in plane strain fracture mode). The tensile properties of Plexiglas II UVA increase linearly throughout the temperature region investigated, with no change in slope with temperature. Similarly,  $\gamma$  for Plexiglas II UVA continues to increase as the test temperature is reduced.

According to the above interpretation, the maximum and consequent decreases in  $\gamma$  as  $T_g$  is approached is the result of increased polymer softening, as indicated by the tensile data.

### Analysis of Data

The mechanism causing the onset of polymer flow has been a subject for speculation for considerable time. There is reason to believe that temperature rise, volume dilation, and a number of molecular factors such as chain entanglement, backbone flexibility, side group size, etc. are involved in the plastic deformation process. There has been considerable support in the literature for the concept of volume dilation as at least a partial cause of yielding in polymers [14-21]. Our tensile yield data appears to further support this concept. The volume dilation theory suggests that as a uniaxial stress is

applied to the polymer, the volume increases according to the relation:

$$\Delta v = (1 - 2\nu) v \epsilon \quad (3)$$

where  $\Delta v$  = increase in polymer volume,  $v$  = volume of the polymer in the unstressed state,  $\nu$  = Poisson's ratio and  $\epsilon$  = uniaxial tensile strain.

If part or all of this volume increase corresponds to polymer free volume generation, then yielding will occur when the free volume generated at the test temperature equals that needed for the polymer molecules to "feel" as though they are at the main glass temperature. This yield process is then dependent on what is referred to as a "pseudo" reduction of the main glass temperature, i.e.  $\Delta T_g = T_g - T_{\text{test}}$ .

There have been at least two theories developed which propose that all of the volume increase from an applied load becomes free volume [16, 18]. Although our yield strain data can be fitted to portions of each of the proposed theories, neither theory successfully predicts the yield strain over the entire temperature range tested.

We propose that not all of the increase in volume during loading becomes free volume, but rather that the dilatation associated with the initial linear elastic response of the polymer does not result in free volume. The total strain to yield is expressed as the sum of two terms, a linear elastic strain,  $\epsilon_e$ , and a non-linear strain,  $\epsilon_v$ , which for lack of a better name will be called a viscous strain. This is shown schematically in Figure 10.

The elastic component is given by  $\sigma_y/E$  where  $E$  is the initial elastic modulus. This is the strain which would be present in the polymer at the yield stress level if no

non-linear deformation occurred. One might visualize this to be the strain in the material if there were no change in the conformation or orientation of the molecules in the glassy state. Rather, the dilation occurs, as it does in simple crystals, with essentially no change in molecular conformation other than an increase in the average spacing between molecules. It is assumed that this elastic deformation does not generate additional free volume.

All of the free volume needed to cause gross yielding is assumed to come from the non-linear increment  $\epsilon_v$  to the total strain. It is emphasized that this viscous flow model represents plastic deformation on a micro level, perhaps the same as in craze formation. It should not be confused with the post-yield homogeneous plastic flow that occurs during cold drawing of a polymer. Thus, the non-linear incremental strain results in a molecular rearrangement that causes an increase in the free volume of the polymer. This latter effect may or may not be accompanied by an additional macroscopic volume change. The relationship between  $\epsilon_v$  and the free volume is shown with the aid of Figure 11. Our definition of free volume is identical to that used by Litt and Tobolsky [16]:

$$\Delta v_{\text{free}} = \Delta v_a - \Delta v_c = \alpha_{ga} \Delta T_g v_a - \alpha_{gc} \Delta T_g v_c \quad (4)$$

where  $\Delta v_{\text{free}}$  = change in specific free volume (cc/g),  $\Delta v_a$  = change in amorphous specific volume (cc/g),  $\Delta v_c$  = change in close-packed specific volume (cc/g),  $\alpha_{ga}$  = volumetric coefficient of thermal expansion in the amorphous glassy state (1/°F),  $\alpha_{gc}$  = volumetric coefficient of thermal expansion in the close-packed glassy state (1/°F),  $v_a$  = amorphous specific volume (cc/g),  $v_c$  = close-packed specific volume (cc/g), and  $\Delta T_g = T_g - T_{\text{test}}$  (°F).

It should be emphasized that although the polymers investigated are highly amorphous, under certain conditions a close-packed or crystalline-like structure can be induced. For instance, the close-packed specific volume of polycarbonate [17] was taken as that value of specific volume obtained from x-ray scattering data on well-annealed polymer samples.

Incorporation of Equation (4) into Equation (3) results in the expression:

$$\epsilon_v = \frac{\Delta v_{\text{free}}}{v_a(1-2\nu)} = (\alpha_{ga} - \alpha_{gc} \frac{v_c}{v_a}) \Delta T_g / (1-2\nu) \quad (5)$$

Thus the yield strain for the polymer is given by:

$$\epsilon_y = \sigma_y / E + (\alpha_{ga} - \alpha_{gc} \frac{v_c}{v_a}) \Delta T_g / (1-2\nu) \quad (6)$$

The elastic component of the yield strain appears to be the same for all four polymers, as shown in Figure 12. Whether this is true for all amorphous polymers is not known, but it is not too likely. Subtraction of the experimental elastic strain in Figure 12 from the experimental yield strain in Figure 8 results in the viscous strain,  $\epsilon_v$ , for the four polymers. As shown in Figure 13, except for the initial 50°F below  $T_g$ , the data appears to be linear with  $\Delta T_g$ . Since at  $T_g$  no viscous strain is needed to cause yielding, the data lines were drawn through zero at  $T_g$ . In accordance with Equation (5), the slope of the line is equal to  $(\alpha_{ga} - \alpha_{gc} v_c / v_a) / (1-2\nu)$ . Table 1 shows the values of  $(\alpha_{ga} - \alpha_{gc} v_c / v_a)$  for the four polymers as determined from Figure 13. The range of values between 2 to  $7 \times 10^{-5}$  (1/°F) appears to be reasonable. The only data available to check the theoretical slope was for polycarbonate [17] and, as seen, the

agreement is excellent. Specific volume measurements on the close-packed state were found at room temperature for polyphenylene oxide and polymethylmethacrylate [16], but a value of  $\alpha_{gc}$  could not be found or reasonably calculated to check the theory for either polymer.

Within 50°F of the main glass transition temperature there appears to be a small deviation in the linearity between  $\epsilon_v$  and temperature, with the experimentally determined values being slightly higher than the linearly extrapolated values. If one includes a temperature dependence in  $v$ ,  $\alpha_{ga}$ , and  $\alpha_{gc}$ , the value of viscous strain would be increased in the region near  $T_g$ , further improving the fit. In particular,  $\alpha_{ga}$  for polymethylmethacrylate has been shown to increase rapidly above 200°F [22]. Poisson's ratio would also be expected to be increasing toward a value of .5 in this temperature region. A more correct form for viscous strain is then:

$$\epsilon_v = \left[ \alpha_{ga}(T) - \alpha_{gc}(T) \frac{v_c(T)}{v_a(T)} \right] \Delta T_g / [1 - v(T)] \quad (7)$$

Although the yield strain data at these temperatures is not sufficiently accurate to merit calculations of this type, Equation (7) will undoubtedly give a better fit of the experimental data.

### Conclusions

The fracture energy of polyphenylene oxide, polysulfone and polycarbonate increases with decreasing temperature to a maximum at roughly 150°F below the main glass transition temperature of the polymers. The maximum appears to be associated with the general changes in viscoelastic properties accompanying the main glass



transition temperature and can be correlated with changes in strength and modulus in this temperature region. A similar maximum in  $\gamma$  with corresponding changes in strength and modulus was not observed for polymethylmethacrylate to as low a temperature as we could study. Presumably polymethylmethacrylate must also show the same type of behavior, but at a much lower temperature than the other three polymers. Below this maximum, the fracture energy decreases almost linearly with decreasing temperature. If one extrapolates the data for three of the polymers to the temperature region of absolute zero, the fracture energy appears to have decreased to the order of the theoretical surface energy.

In addition, the volume dilation theory has been further supported by our polymer yield strain data. Although no definite mathematical analysis involving strain at the crack tip has been developed as yet, in light of the correlation of the transitions in the fracture energy with the tensile data, the same equations which describe the onset of yielding in a tensile sample may also be applicable to the crack tip yielding process. If this is true, further work of this nature should provide a method for predicting the fracture energy from tensile stress-strain properties.

#### Acknowledgment

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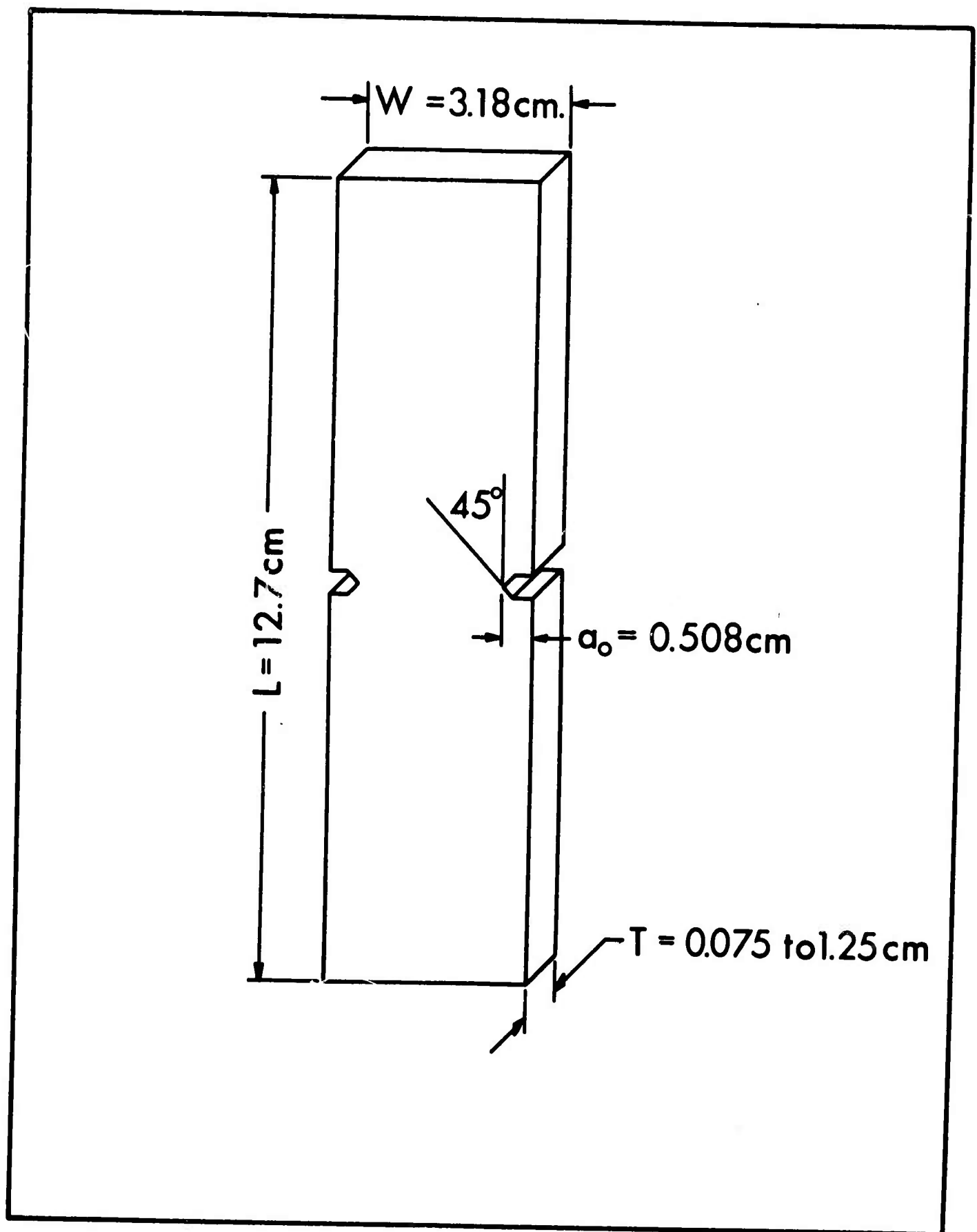


Figure 1. SAMPLE GEOMETRY FOR FRACTURE ENERGY TEST

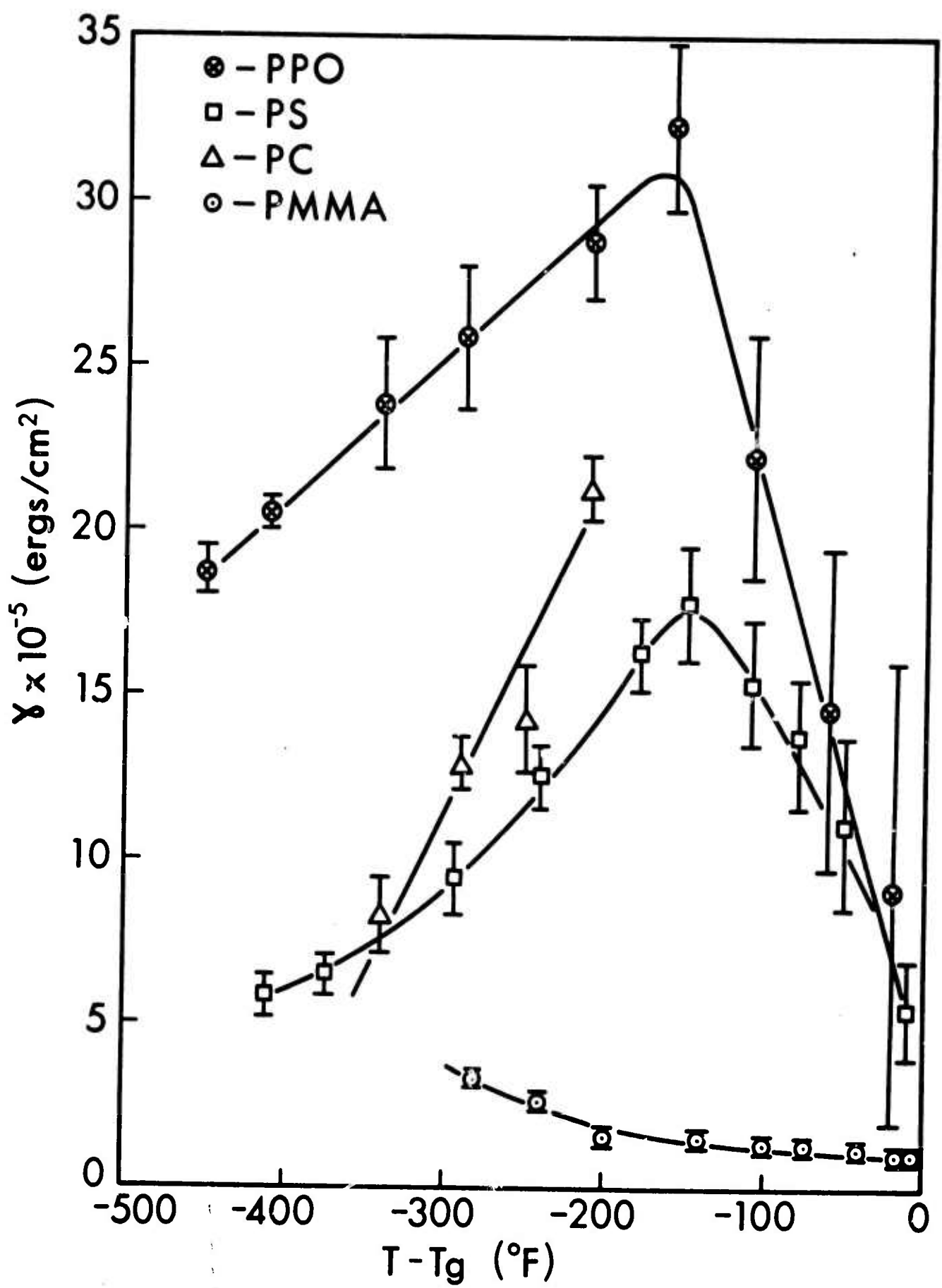


Figure 2. POLYMER FRACTURE ENERGY AS A FUNCTION OF TEMPERATURE

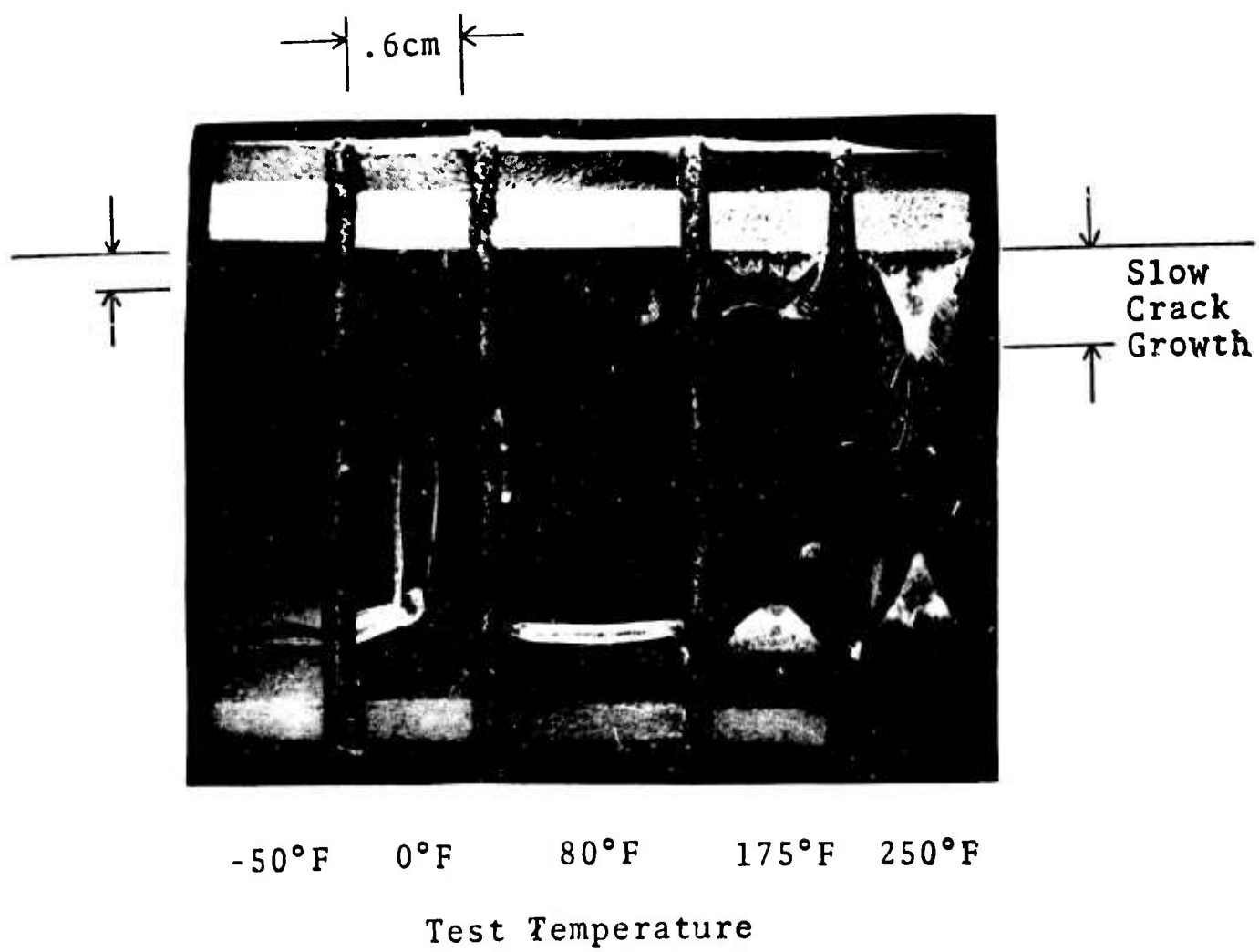


FIGURE 3      POLYCARBONATE FRACTURE SURFACES

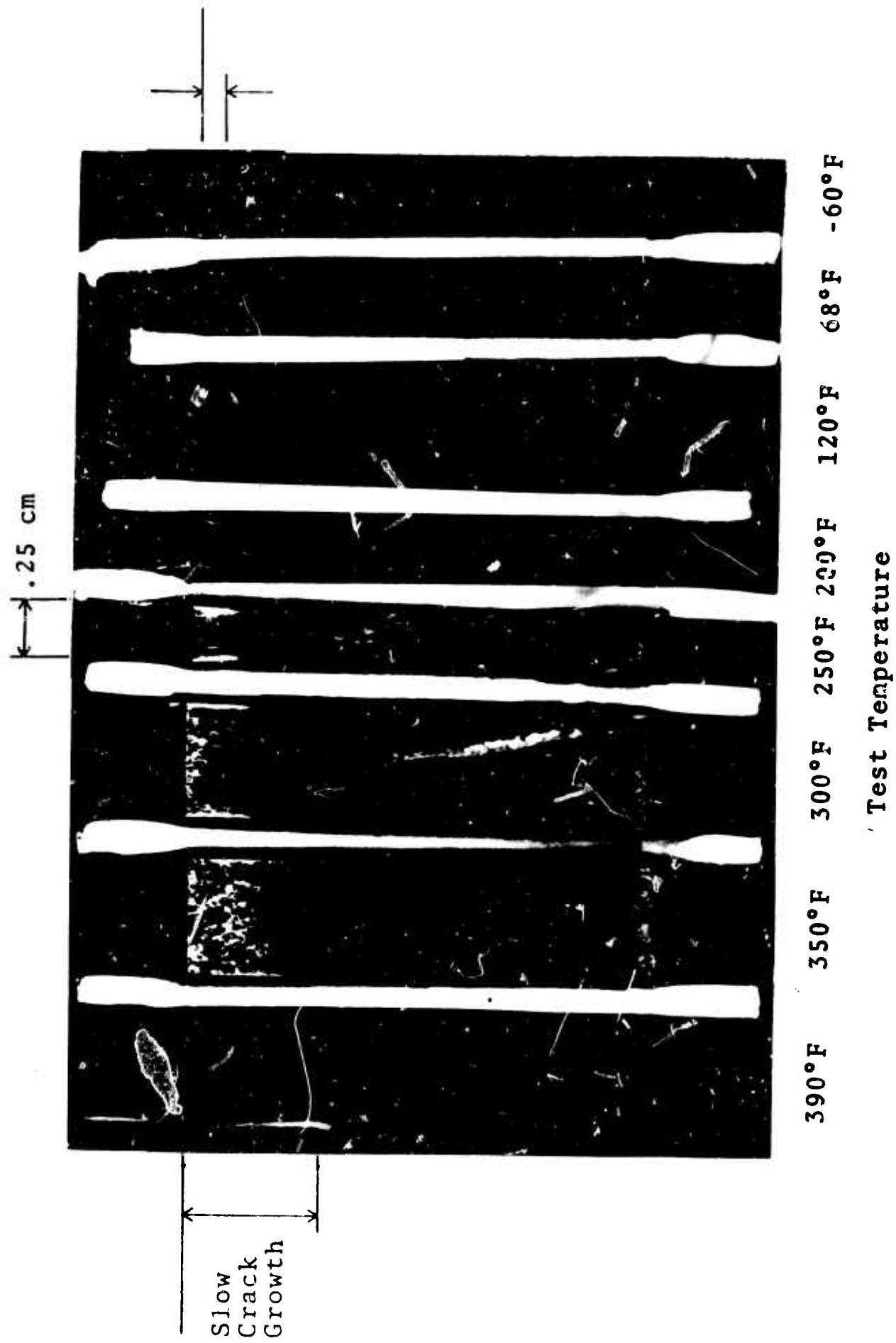


FIGURE 4 POLYPHENYLENE OXIDE FRACTURE SURFACES

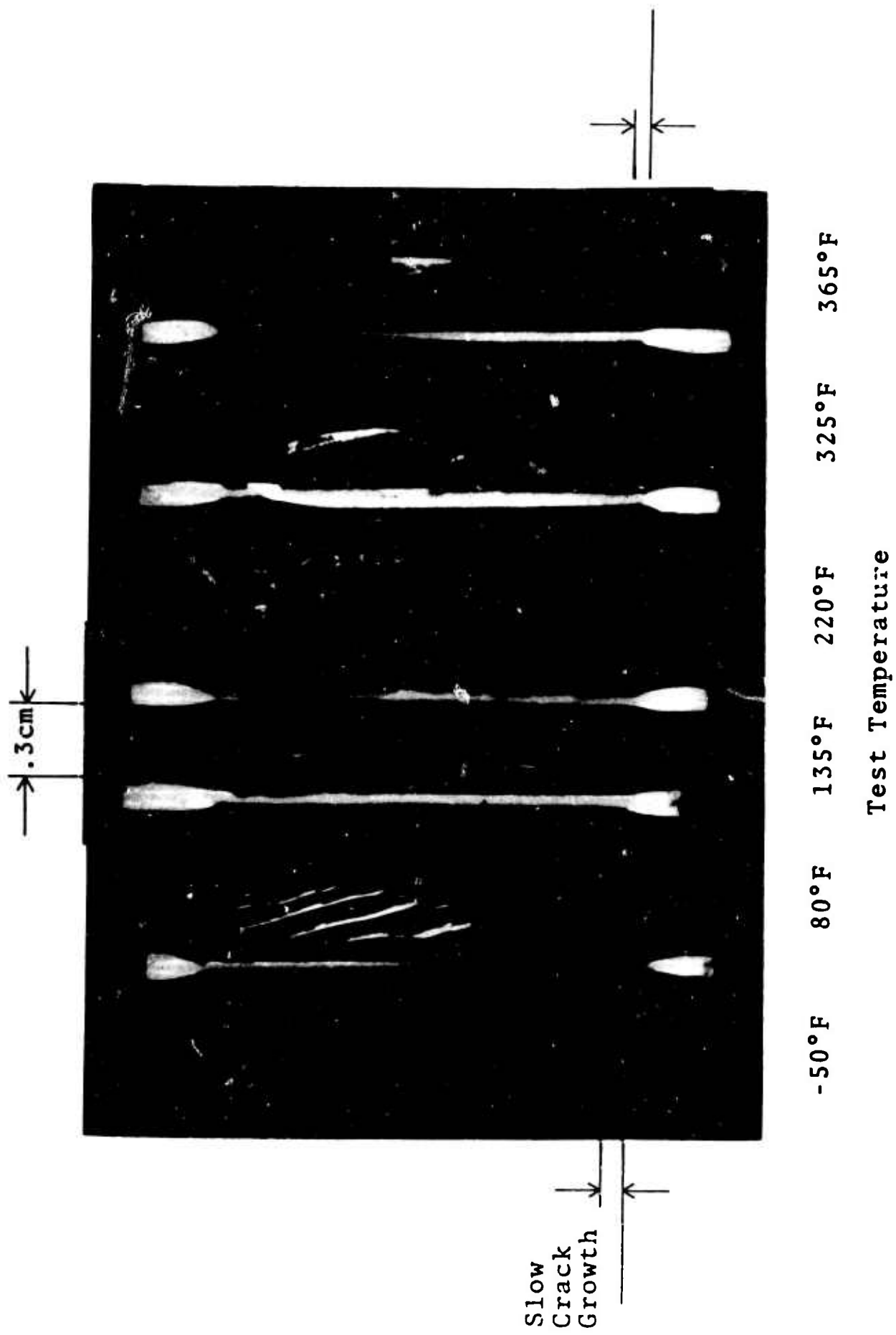
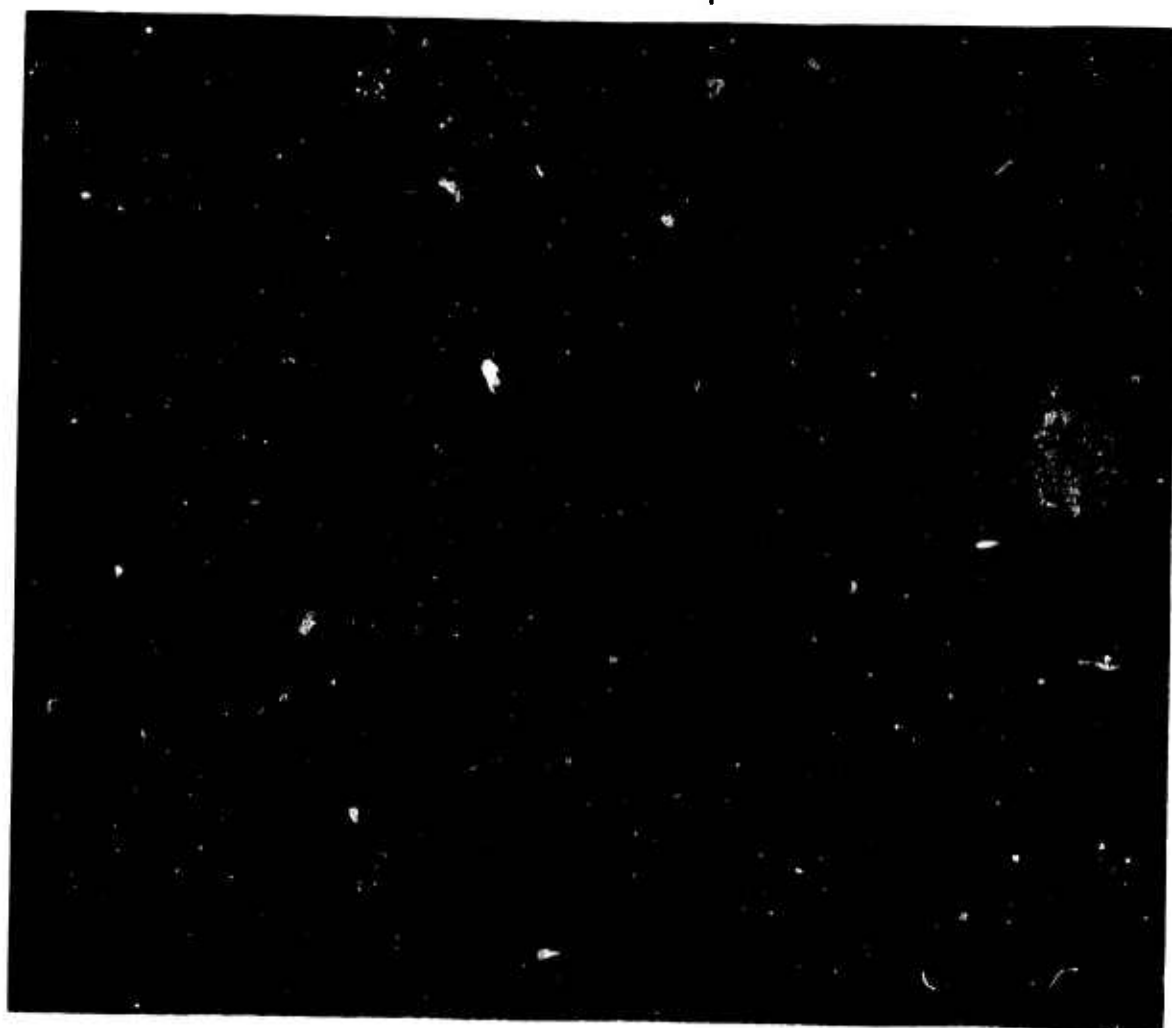


FIGURE 5 POLYSULFONE FRACTURE SURFACES



Negligible Slow Crack Growth

→ .63 cm ←



150°F

200°F

215°F

219°F

221°F

Test Temperature

FIGURE 6

POLYMETHYLMETHACRYLATE FRACTURE SURFACES

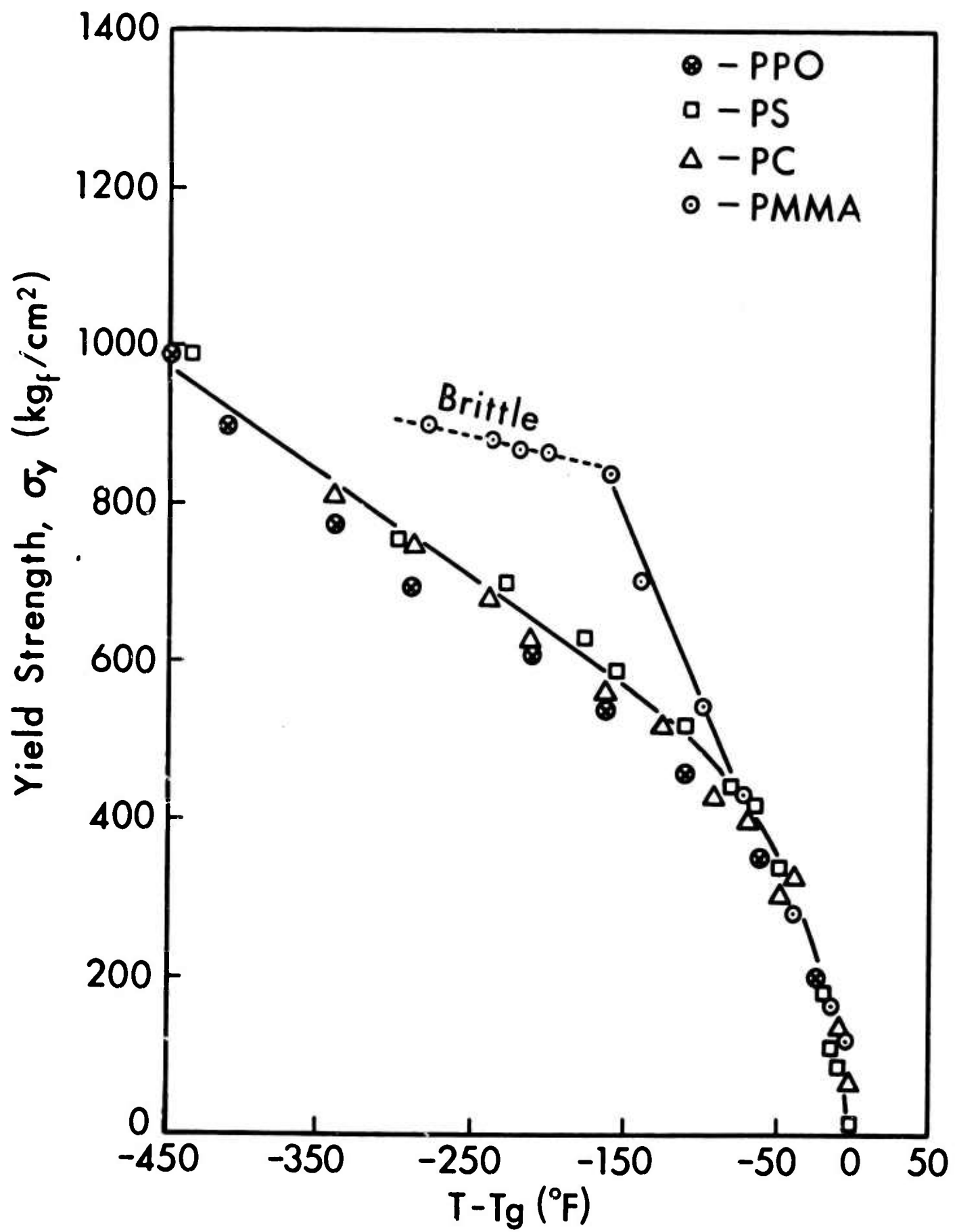


Figure 7. EFFECT OF TEMPERATURE ON POLYMER YIELD STRENGTH

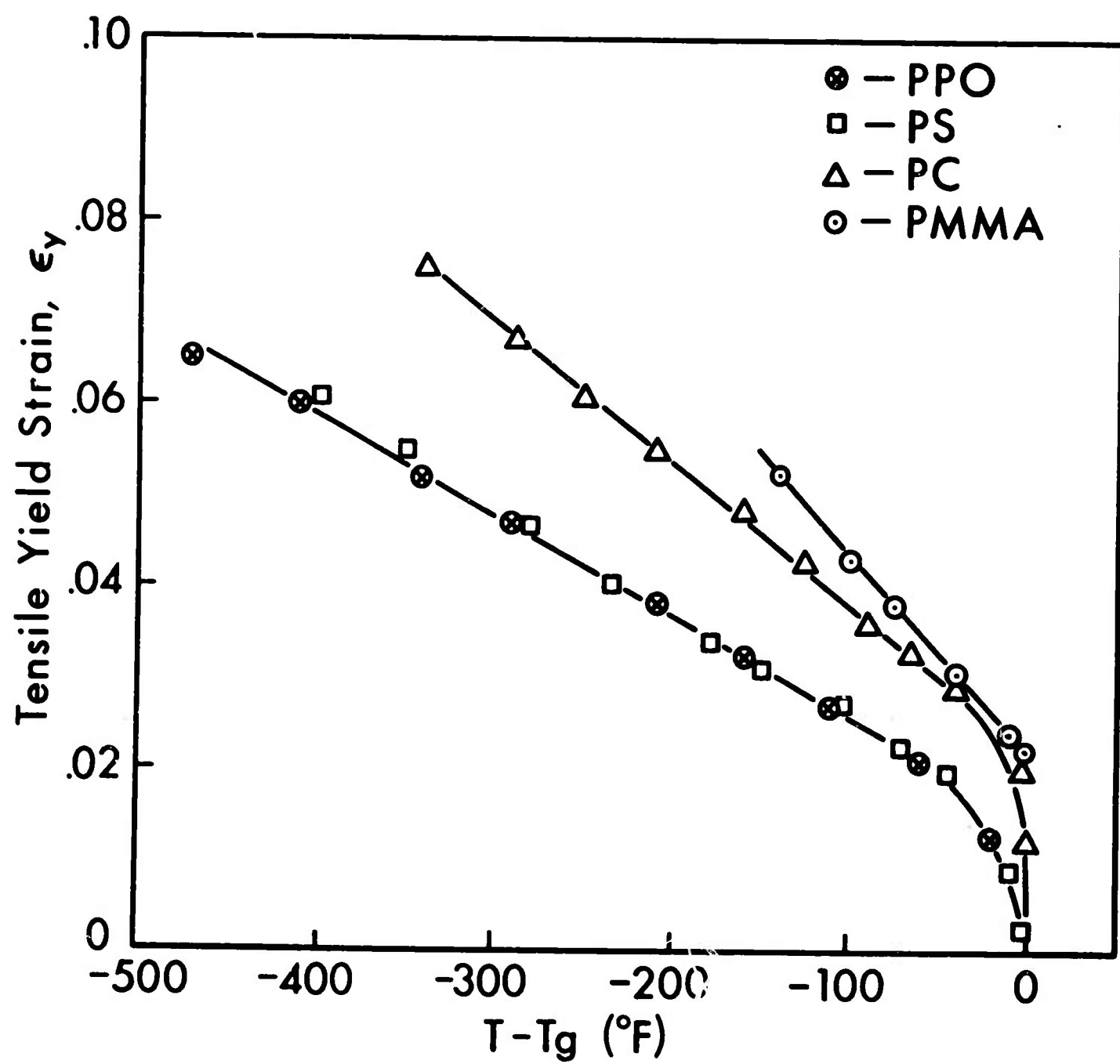


Figure 8. EFFECT OF TEMPERATURE ON POLYMER YIELD STRAIN

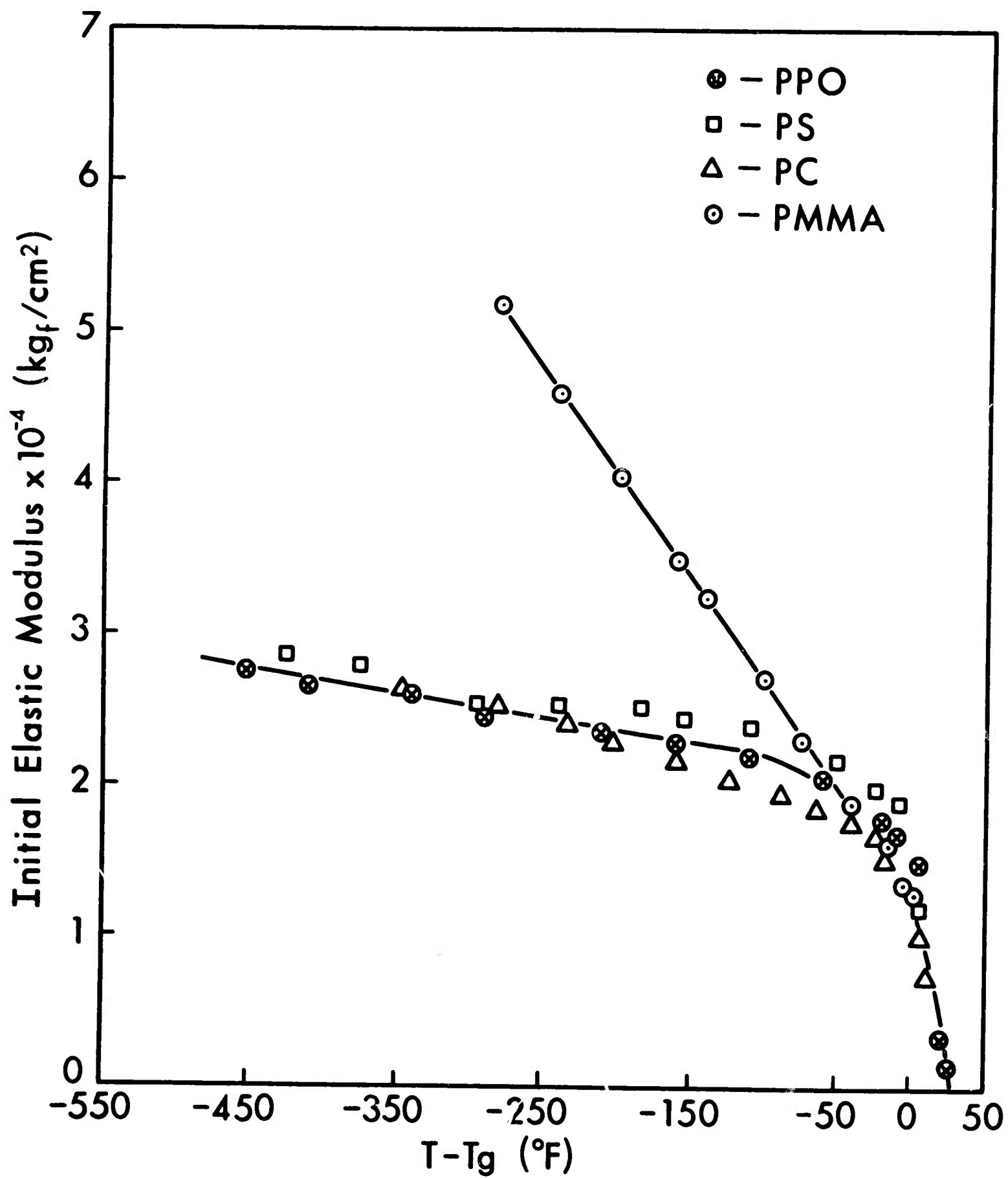


Figure 9. EFFECT OF TEMPERATURE ON POLYMER INITIAL MODULUS

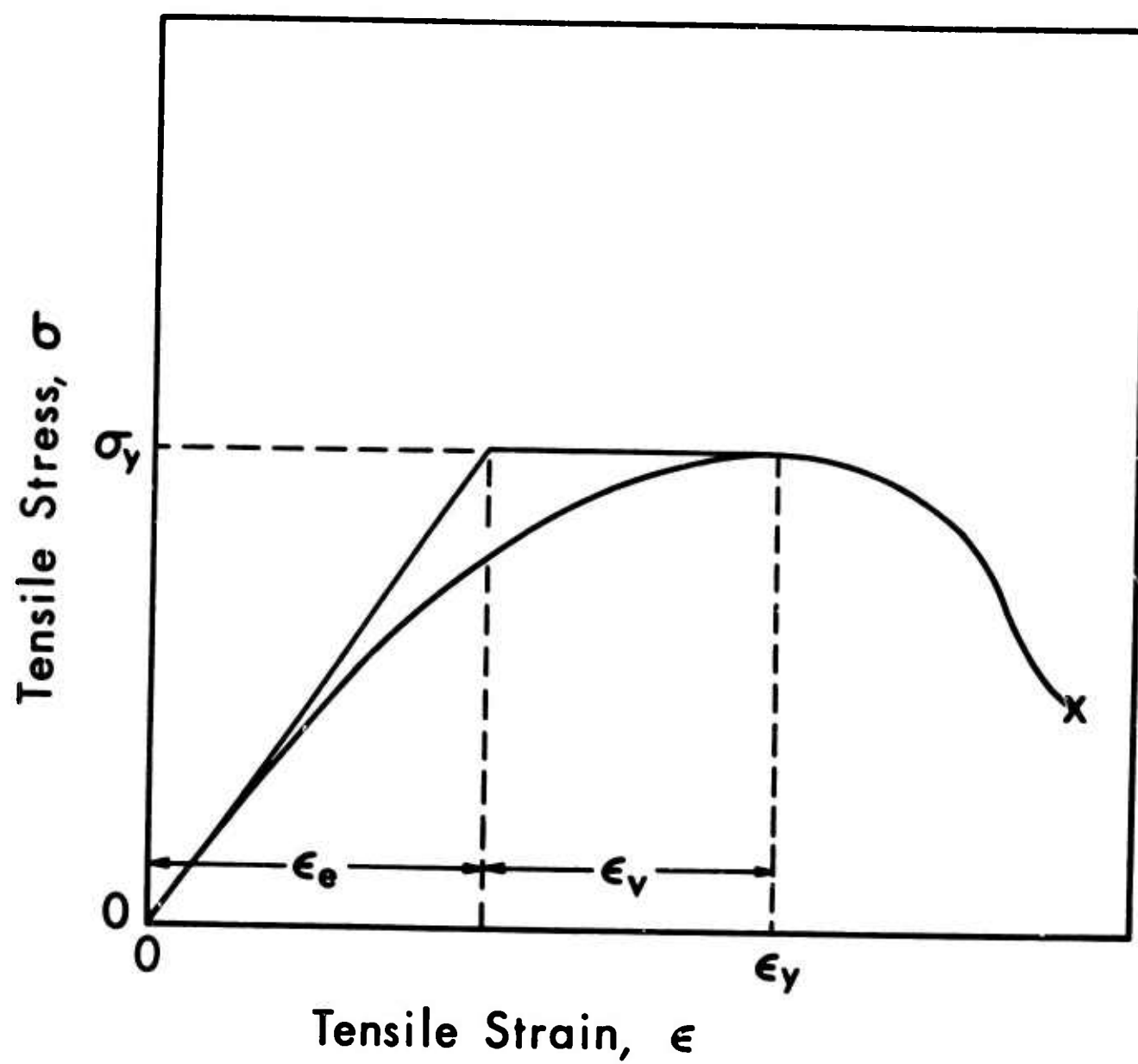


Figure 10. SCHEMATIC DIAGRAM OF THE PROPOSED ELASTIC AND VISCOUS STRAINS

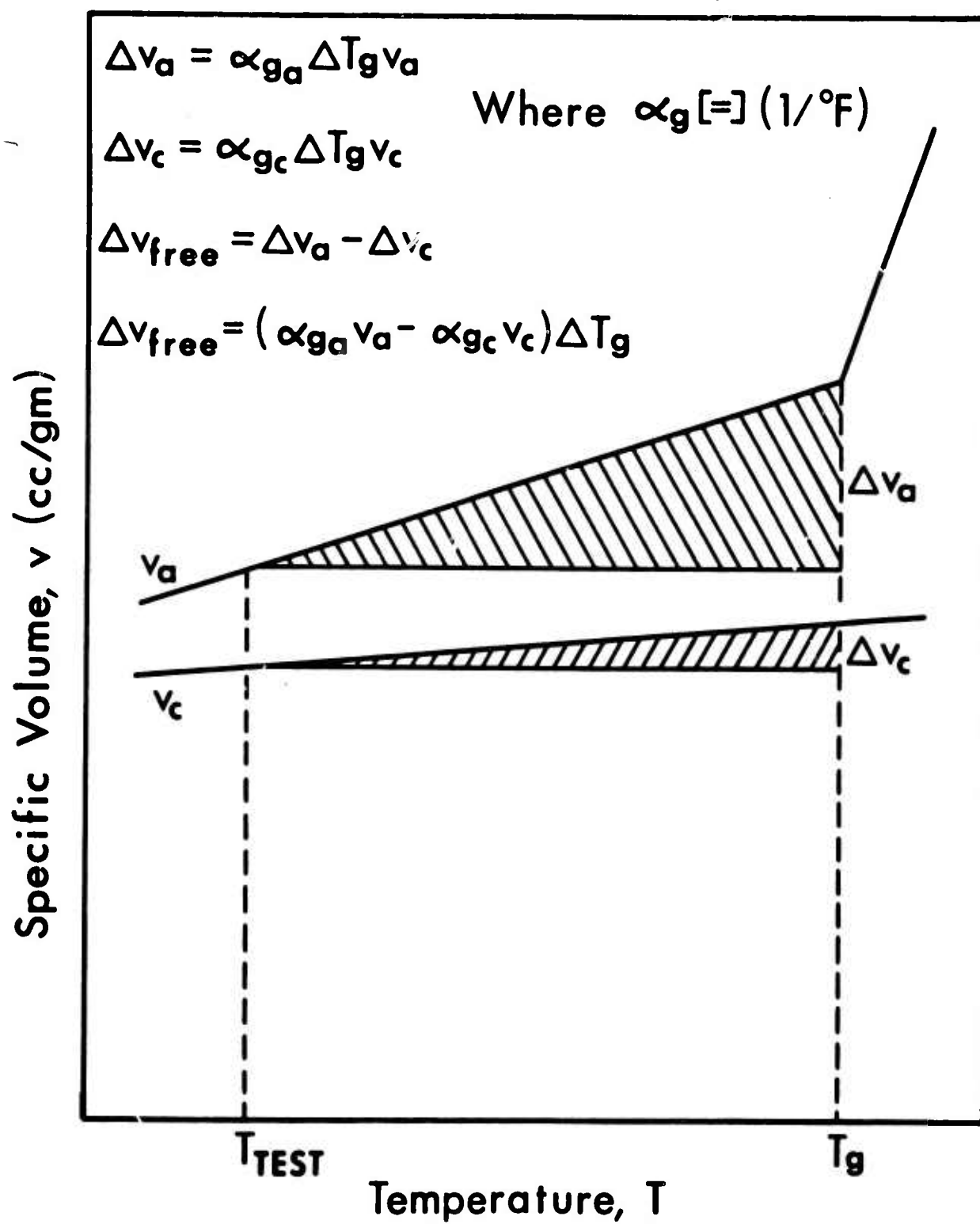


Figure 11. POLYMER FREE VOLUME DEFINED TO INCLUDE CHANGES IN AMORPHOUS AND CLOSE-PACKED VOLUME

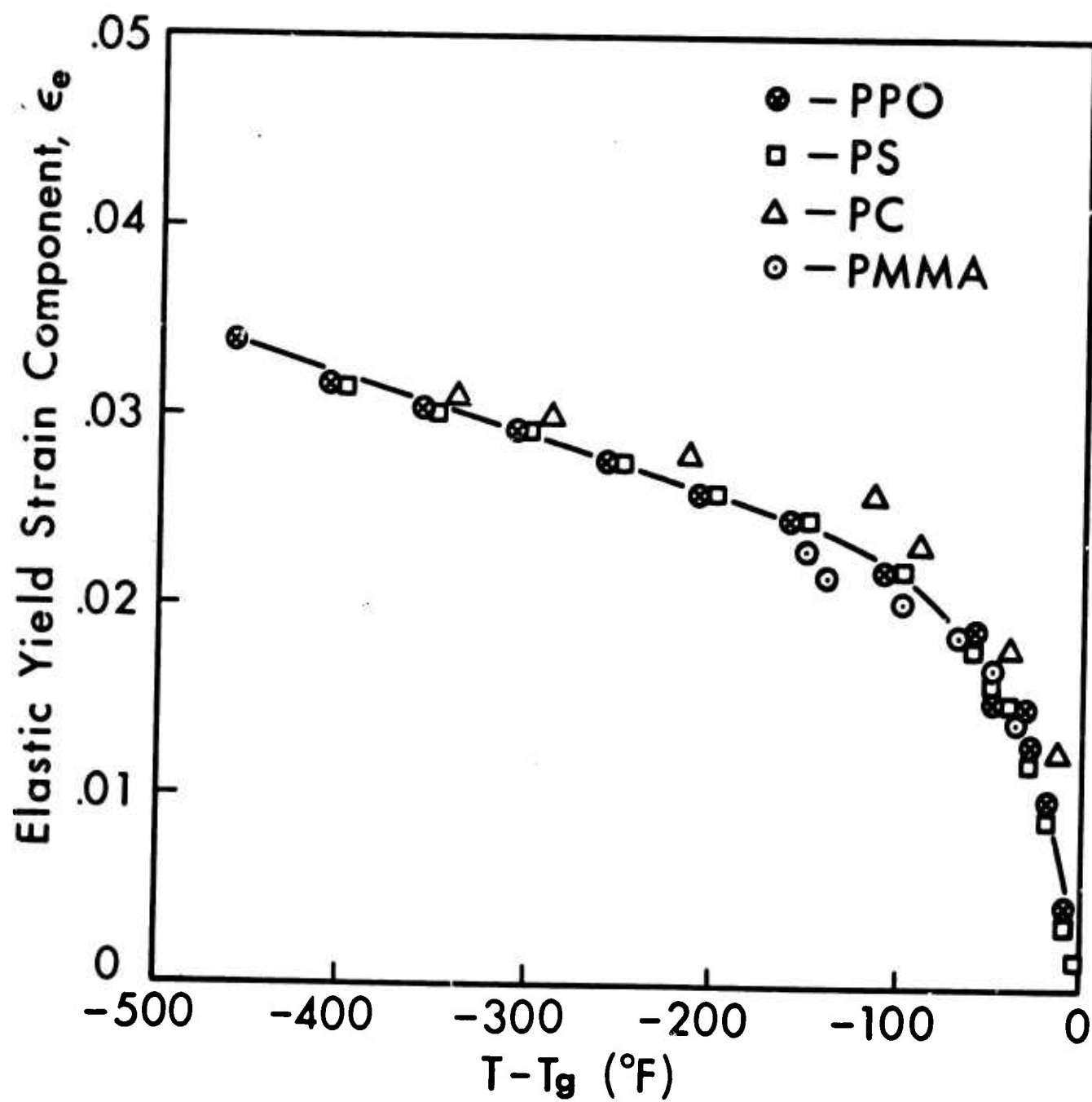


Figure 12. VARIATION IN THE ELASTIC STRAIN WITH TEMPERATURE

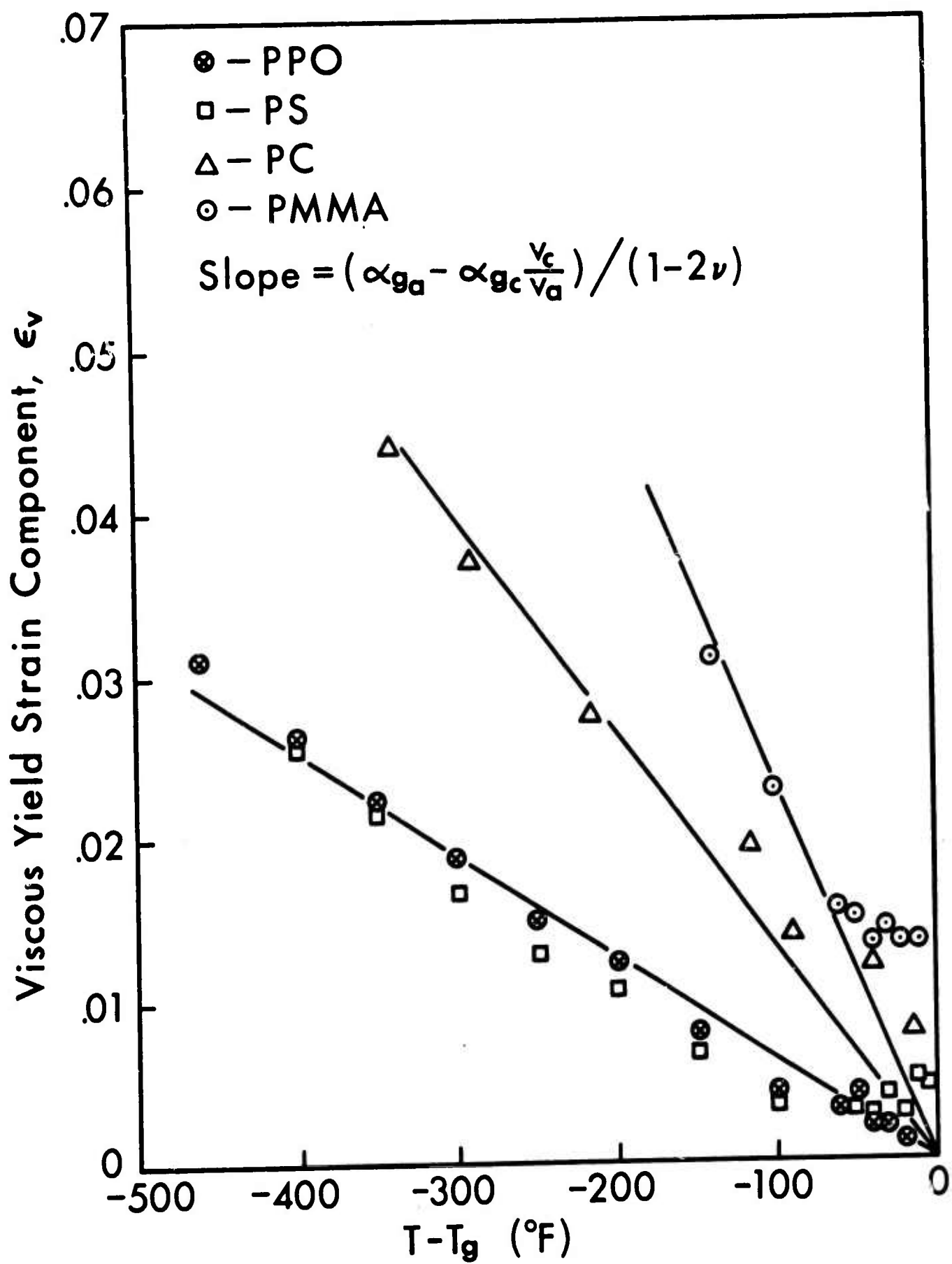


Figure 13. VARIATION IN THE VISCOUS STRAIN WITH TEMPERATURE



Table 1. COMPARISON OF THE PREDICTED AND MEASURED VALUES OF  $(\alpha_{ga} - \alpha_{gc} \frac{v_c}{v_a})$

Polymer	From Figure 13 $\alpha_{ga} - \alpha_{gc} \frac{v_c}{v_a} (1/^\circ\text{F})$	Measured Value $\alpha_{ga} - \alpha_{gc} \frac{v_c}{v_a} (1/^\circ\text{F})$
PPO	$2 \times 10^{-5}$	—
PS	$2 \times 10^{-5}$	—
PC	$3.4 \times 10^{-5}$	$3.6 \times 10^{-5} (16)$
PMMA	$7 \times 10^{-5}$	—

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<p>The brittle fracture properties of polyphenylene oxide, polysulfone, polycarbonate, and polymethylmethacrylate thermoplastic polymers were investigated over a wide range of temperatures. Fracture energy measurements were made using double edge-notched tensile samples. Tensile strength, tensile strain, and initial elastic modulus were measured for calculation of the fracture energy and further analysis of the polymer behavior.</p> <p>It was found that mechanical transitions in the tensile properties corresponded reasonably well with transitions in the fracture energy in the temperature range investigated. Fracture surface photographs permitted visual analysis of the fracture process. It was found that the roughest fracture surface corresponded to the maximum in the fracture energy for a given polymer.</p> <p>A theory for prediction of polymer tensile yield strain is presented, based on the volume dilation concept. The implications of this theory are discussed in terms of the crack tip flow process leading to brittle fracture.</p>			

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